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TENTATIVE PROCEDURE ANALYZING PESTICIDE RESIDUES IN SOLID WASTE

NATIONAL ENVIRONMENTAL RESEARCH CENTER

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Tentative Procedure Analyzing Pesticide Residues in Solid Waste

National Environmental Research Center
Office of Research and Monitoring
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

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Tentative Procedure Analyzing Pesticide Residues In Solid Waste

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NATIONAL ENVIRONMENTAL RESEARCH CENTER
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The Solid Waste Research Laboratory of the National Environmental Research Center, Cincinnati has reviewed this report and approved its publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

ABSTRACT

Because of the concern about pesticide residues in the environment, a procedure to analyze solid waste samples for pesticides was developed. Nine samples of solid waste from municipal refuse; incinerator emissions, residue, and fly ash; and compost were prepared, extracted, and cleaned up with column and thin layer chromatography for analysis by gas liquid chromatography. The cleanup methods were carefully selected to yield reproducible results upon final GLC analyses.

When the chromatograms from the solid waste samples were compared with standards, lindane, o,p'-DDD, p,p'-DDT, chlordane, o,p'-DDE, and p,p'-DDE were identified as possible contaminants of municipal refuse and the products of its disposal.

FOREWORD

To find, through research, the means to protect, preserve, and improve our environment, we need a focus that accents the interplay among the components of our physical environment--the air, water, and land. The missions of the National Environmental Research Centers--in Cincinnati, Research Triangle Park, N. C., and Corvallis Ore.--provide this focus. The research and monitoring activities at these centers reflect multidisciplinary approaches to environmental problems; they provide for the study of the effects of environmental contamination on man and the ecological cycle and the search for systems that prevent contamination and recover valuable resources.

Man and his supporting envelope of air, water, and land must be protected from the multiple adverse effects of pesticides, radiation, noise, and other forms of pollution as well as poor management of solid waste. These separate pollution problems can receive interrelated solutions through the framework of our research programs--programs directed to *one* goal, a clean livable environment.

This publication, published by the National Environmental Research Center, Cincinnati, reports on work from this center. The problems created by pesticide residues in the environment are of concern to everyone. We, with publication of *Tentative Procedure: Analyzing Pesticide Residues in Solid Waste*, offer researchers a starting point for future research in this particular area of solid waste.

ANDREW W. BREIDENBACH, Ph. D.
Director, National Environmental
Research Center, Cincinnati

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CONCLUSIONS

By employing the tentative procedures developed here to analyze solid waste for pesticide residues, the results indicated the presence of various pesticides in the solid waste samples.

With the use of these procedures as a basis for future research, it is hoped that further refinements will be developed for more exacting systems.

TENTATIVE PROCEDURE: ANALYZING PESTICIDE RESIDUES IN SOLID WASTE

INTRODUCTION

Public concern has grown recently over the spread of pesticides throughout the world. The persistence of DDT and its metabolites in the ecosystem is the cause of most concern. Pesticide sales increased 84% in the United States from 1955 to 1965. The production in 1965 was 875 million pounds, which amounted to $250 million in sales. With the population of the United States now increasing at the rate of 3 million persons each year, past experience has indicated that we may expect a correspondingly large increase in production of pesticides. This increased production will increase pesticide residues throughout the environment and consequently in solid wastes (1).

The main objective of this project undertaken at the Solid Waste Research Laboratory of the National Environmental Research Center, Cincinnati, was the development of a procedure for the analysis of pesticides in solid waste samples. The complex and heterogeneous nature of solid waste samples makes pesticide analysis an extremely challenging task. The cleanup methods utilized were carefully selected to yield reproducible results upon final analysis by gas-liquid chromatography.

EXPERIMENTAL

A. Sample Preparation

Nine solid waste samples were selected for analysis to determine pesticide residues in the waste and waste byproducts. Table 1 lists the sample source and size.

After collection, the municipal refuse (Sample 1) was ground in a large hammermill to approximately a 1- by 1-inch size. Following this, a grab sample was ground in a Wiley Mill until it could pass through a 2-mm screen. The sample was then dried in a laboratory oven at 105°C for several hours. After drying, the

TABLE 1. SOLID WASTE SAMPLES ANALYZED

Sample No.	Source	Amount extracted
1	Municipal refuse	50.9 grams
2	Incinerator stack condensate	250 ml
3	Trapped gaseous incinerator emissions	300 ml*
4	Incinerator residue	90.1 grams
5	Incinerator residue	254.0 grams
6	Incinerator fly ash	115 7 grams
7	Incinerator fly ash	87.8 grams
8	0-Day compost	50.1 grams
9	42-Day compost	69.3 grams

*This 300 ml was the collecting agent for almost 19 cubic feet of gaseous emissions.

sample was weighed into preextracted soxhlet thimbles for pesticide extraction.

The condensate and trapped gaseous emissions (Samples 2 and 3) were obtained from an experimental high-temperature (maximum temperature of 2,500°F), low-capacity combustion unit. A sample probe was placed in the stack, and a portion of the gaseous emissions was passed through this probe. These emissions were routed through a series of water-cooled condensers, then through a gas-diffusion bubbler immersed in ethylene glycol. The purpose of the ethylene glycol was to extract any organic material, especially pesticides, that had been volitalized in the combustion process. Eventually a water condensate began building in the condensers; this was collected and utilized as a solid waste processing sample along with the ethylene glycol material. These two samples were extracted with the use of the procedure described by FWPCA(2).

The residue and fly ash samples (Samples 4-7) were prepared according to the method described by Cohen and Allen (3) and stored in the laboratory at room temperature until analyzed. The procedure of Cohen and Allen involves various sorting, grinding, quartering, and drying procedures to reduce the sample volume and particle size to useful working dimensions. In final treatment, the material used in this study was pulverized so the particles would pass a 60-mesh sieve.

The compost samples (Samples 8 and 9) were initially ground, mixed, and dried at 100°C to a constant weight at the compost plant in Johnson City, Tennessee. On arrival, the large pieces of glass, ceramics, metals, and rocks were removed manually, and the samples were then ground in a Wiley Mill until they could pass a 2-mm screen. All of the compost samples originated from the same windrow, and each sample represented a composite of many small grab samples collected at various sites along the windrow.

B. Extraction

All samples, except 2 and 3, were solid and were extracted with the use of a soxhlet apparatus that contained a preextracted 43- by 123-mm thimble that was attached to a 300-ml, round-bottom flask. The extracting solvent was 9 parts hexane to one part acetone. After all of these solid samples were extracted for at least 12 hours, the solvent was evaporated to dryness in a vacuum oven at room temperature.

Treatment of samples 2 and 3 involved two extractions, one with 150 ml of 15% ethyl ether in hexane and the other with 150 ml of hexane only. These extracts were combined and then were passed through a column of anhydrous sodium sulfate and evaporated to dryness in a vacuum oven at room temperature.

C. Cleanup – Column Chromatography

After evaporation of the extracts, the residues were redissolved in a small quantity of hexane. A 15-gram charge of Florisil (which had been stored in an airtight container at 130°C) was added over a ½-inch layer of anhydrous sodium sulfate in a 300-mm-long Pyrex glass column with a 25-mm O.D. After tapping the Florisil into the column, another layer of sodium sulfate (¾-inch deep) was added to the top. The column was allowed to cool to room temperature and was preeluted with about 75 ml of hexane. This 75 ml was discarded, and just prior to exposure of the top layer of sodium sulfate by the eluting solvent, the sample extract was quantitatively transferred into the column by decantation and subsequent hexane washings. The elution rate was adjusted so that it did not exceed 5 ml per minute. The complete column cleanup procedure involved two separate solvent systems: the first elution was performed with 200 ml of 6% ethyl ether in hexane, the second, with 200 ml of 15% ethyl ether in hexane(2). These eluates were collected and evaporated to dryness for thin layer chromatography cleanup.

D. Cleanup – Thin Layer Chromatography (TLC)

The residues of the evaporated eluates from the column cleanup were redissolved in 1 ml of hexane for cleanup by TLC. The TLC system described by Lichtenstein *et al.*(4) was utilized at the final cleanup prior to gas-liquid chromatography (GLC). The TLC system employed activated aluminum oxide plates developed in a solvent system 99% heptane and 1% acetone. Standards and at least 100 µl of the sample were put on the plate. The 20- by 20-cm plate was then developed to 15 cm and allowed to air dry. The standards were then sprayed with a solution of Rhodamine B (0.1 mg/ml in ethanol), with the sample portion being covered so that it was not sprayed. The sprayed portion was allowed to thoroughly dry (about 5 minutes) and was then viewed under short wavelength UV light.

The standards were marked, and the area of sample corresponding to the area of standard was

collected from the plate. This material was placed in a 15-ml centrifuge tube and extracted three times with 10-ml portions of hexane. After the hexane was added, the material was thoroughly mixed and then centrifuged to yield a clear supernatant. This liquid was collected and evaporated to 2 ml volume. One ml was removed from each sample, transferred to a separate vial, evaporated to dryness, and sent to the Perrine Primate Laboratory for GLC analysis. The remaining 1 ml was analyzed at Analytical Quality Control Laboratory (1014 Broadway, Cincinnati). Figure 1 shows the TLC plate for standards and samples.

E. Analysis – Gas-Liquid Chromatography

Four instruments were employed for analysis: A, B, C, and D.

Instrument A was operated with the following parameters:

G. C.,	Barber Colman Series 5000 floor model
Column,	6-foot U-tube, 4 mm I.D., glass
Packing,	10% DC-200 on Anakrom ABS 80/90 mesh
Detector,	tritium foil
Gas flow,	100 ml/min N_2
Column temperature,	200°C
Detector temperature,	212°C
Injector temperature,	211°C

Instrument B was operated with the following parameters:

G. C.,	Micro Tek 160
Column,	8-foot tube, ¼-inch O.D., aluminum
Packing,	3% DC-200 + 5% QF-1 on Gas Chrom Q 80/100 mesh
Detector,	tritium foil
Gas flow,	80/min N_2
Column temperature,	202°C
Detector temperature,	202°C
Injector temperature,	220°C

Instrument C was operated with the following parameters:

G. C.,	Micro Tek 220
Column,	6-foot U-tube, 4 mm I. D., glass
Packing,	1.5% OV-17 + 1.95% QF-1 on Chromosorb W. H. P., 100/120 mesh
Detector,	tritium foil
Gas flow,	100 ml/min N_2
Column temperature,	188°C
Detector temperature,	200°C
Injector temperature,	225°C

Instrument D was operated with the following parameters:

G. C.,	Micro Tek 220
Column,	6-foot U-tube, 4 mm I. D., glass
Packing,	Chromosorb W. H. P., 80/100 mesh
Detector,	tritium foil
Gas flow,	65 ml/min N_2
Column temperature,	195°C
Detector temperature,	205°C
Injector temperature,	225°C

DISCUSSION

A. Samples

The municipal refuse (Sample 1) was a grab sample from a 10-cu-yd packer truck collecting from residential sources in St. Bernard, Ohio, during the summer when lawn clippings are high.

The condensate and trapped gaseous emissions (Samples 2 and 3) were collected from an experimental, high-temperature, low-capacity incinerator and represented approximately 19 cu ft of gaseous emissions trapped.

The incinerator residue and fly ash samples were collected while personnel from Federal solid waste activities evaluated municipal incinerators in Medina, Pennsylvania (Samples 4 and 6), in Greenwood, South Carolina (Sample 5), and in New Orleans, Louisiana (Sample 7).

Samples 8 and 9 came from the joint U.S. Public Health Service–Tennessee Valley Authority Composting Project at Johnson City, Tennessee. Both samples came from the same windrow: Sample 8 represented 0-day compost, closely resembling raw refuse, and Sample 9 represented the end product of the composting process, 42-day-old compost.

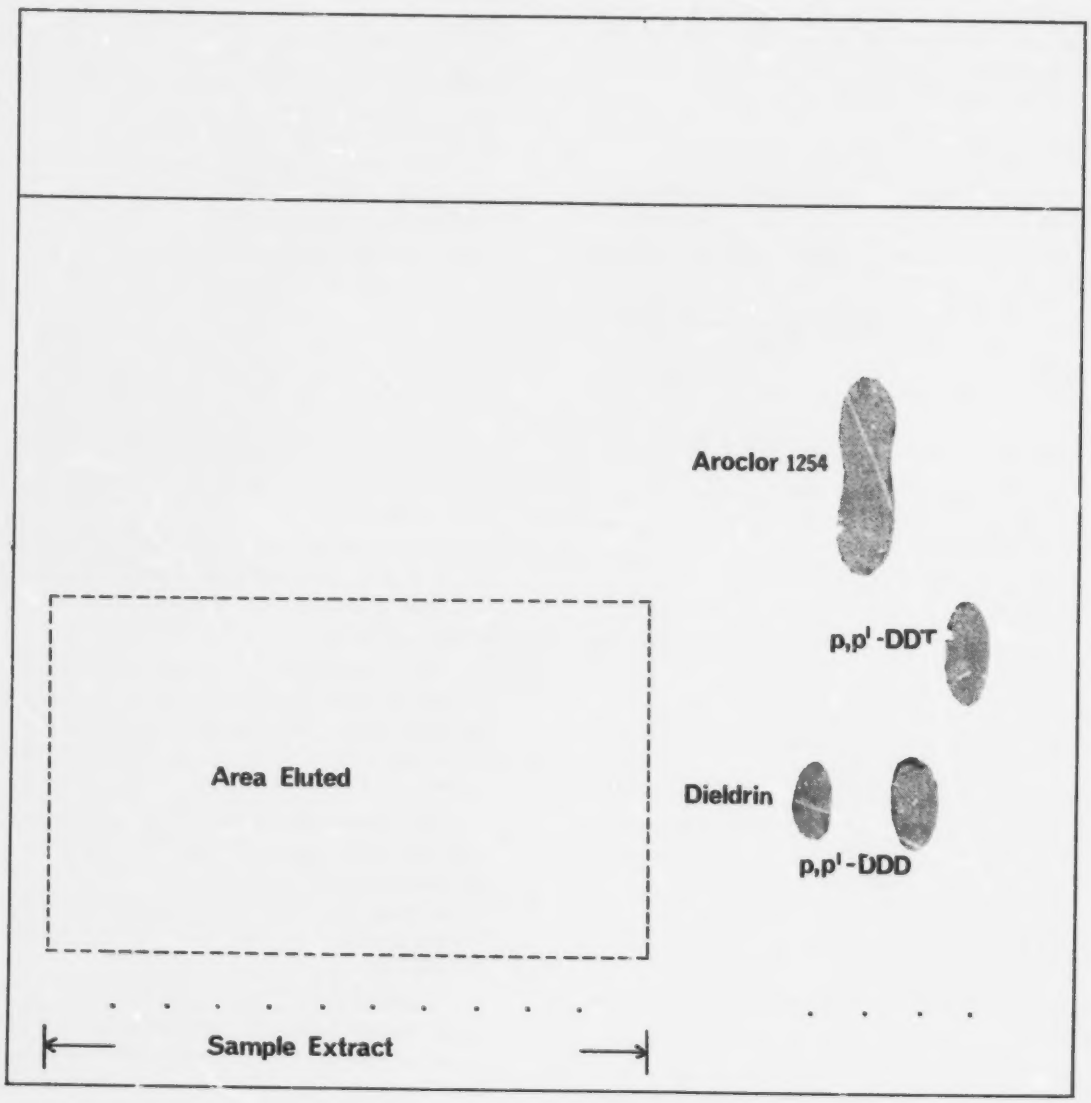


Figure 1. Thin layer chromatogram of pesticide standards and sample showing area eluted for further analysis. (Reduced 20%.)

B. Sample Preparation

For all samples (except 2 and 3), a complex process of preparation was followed. Care was taken to produce a homogeneous sample, and the samples were closely examined to see that large pieces of glass, wood, metal, ceramics, etc., had been removed before grinding. The sample used in the laboratory was the end result of many hours of tedious labor, both in collection and preparation, and was representative of a large volume of solid waste and solid waste disposal processes.

C. Extraction

A search of the literature yields many possible extracting solvents, any of which would perform adequately. The 9:1 hexane-acetone solution was selected because it had previously been used on solid samples, particularly bottom muds and soil. and because time did not permit experimenting with various solvents.

D. Column Chromatography

The activated Florisil column was selected after a column of Florisil:Celite was studied and found unacceptable for this project. A review of the literature indicates activated Florisil is widely accepted for this use.

E. Thin Layer Chromatography

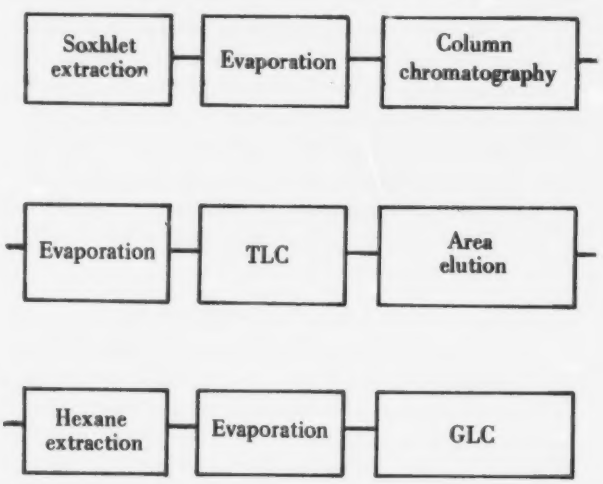
The immediate goal of the TLC system was to remove the polychlorinated biphenyls (PCB's) that interfere with pesticide analysis. The selected TLC system did this and also removed some residual color from the extracts, along with oils and grease that came through during the column cleanup step.

A TLC absorbent of silica gel was studied with the use of standards; it was found that PCB's interfered with the pesticides because both traveled similar distances during development.

F. Analysis

An alternative source of final analysis was sought because of the constant operational problems encountered with Instrument A. Instruments B, C, and D are frequently used for pesticide analysis and yield highly reproducible results. Because the detectors in instruments B, C, and D do not become contaminated as easily as that of Instrument A, fewer operational problems are encountered.

Schematically, the sample extraction and cleanup procedures before GLC are



SUMMARY

A. Sample Preparation

A method for preparing a large number of solid waste samples previously investigated by the Solid Waste Research Laboratory was found to be satisfactory.

B. Extraction

The solvent system of hexane–acetone recovered more than 95% of p,p'-DDT when added to a refuse sample similar to Sample 1; however, some research is needed to investigate other systems that could possibly be oriented to yield an extract lower in oils and greases that will still yield the desired pesticides.

In the literature, many systems are given for extracting pesticides. Alternative solvent systems were not investigated, primarily because of time limitations.

C. Cleanup – Column Chromatography

Although the column cleanups were grossly overloaded because of the large amount of extractables from the soxhlet extractive step, they did remove much of the color, oil, and grease. We believed that activated Florisil would suffice here because most investigators of pesticides use the Florisil system.

D. Cleanup–Thin Layer Chromatography

Again, time did not permit the extensive investigation of TLC systems. A system, found in the literature, that separated PCB's from pesticide residues was used because this was considered the most important function of this cleanup as far as pesticide analysis was concerned.

Since new systems are continually being added to the literature, we suggested that in the future other TLC systems be investigated; they may yield better results than those obtained in this project.

E. Analysis–Gas-Liquid Chromatography

The last step of the experimental procedure is the GLC analysis. The need for a good sound instrument becomes imperative when dealing with pesticide residue analysis. The chromatograms obtained from instrument B (Figures 2-4) show the results of a mixed pesticide standard sample, a chlordane standard, and the results of 0-day compost sample. By comparing the results obtained from the standards with those from the 0-day compost sample, we concluded the compost sample contained lindane, p,p'-DDT, and chlordane. Other peaks were observed, but identification could not be made at that time.

When a more sensitive instrument (C) was used with the same comparison procedure, the results indicated the possible presence of lindane, p,p'-DDE, o,p'-DDE, p,p'-DDT, and chlordane (Figure 5).

The chromatograms obtained from Instrument D (Figures 6 and 7) show the many chlordane peaks (Figure 6) and the many peaks obtained from the 0-day compost. Some possible identifications in Figure 7 are lindane, o,p'-DDD, p,p'-DDT, and chlordane. Once again, the identifications were made by comparing pesticide standards.

Instrument A failed to function during most of this project; thus, the final analyses were performed on instrumentation better suited for pesticide analysis.

The choice of columns and column packing (liquid phase and solid support) is very important for accurate separation of pesticide residues; therefore, we recommend that more research be conducted in this particular phase of final analysis.

The results of all the chromatographic findings are shown in Tables 2, 3, and 4. As indicated in these tables, no pesticides of any significance were identified from the types of samples analyzed. Table 2 lists standards for the several pesticides run on each instrument. The calculated relative retention time for each standard relative to an aldrin standard, the various samples, and the instrument that detected the possible presence of each pesticide are also listed.

Tables 3 and 4 list, by instrument and sample, which pesticides were indicated from the chromatograms obtained from each instrument for each sample. As can be seen, at least two of the three instruments indicate the presence of chlordane for several samples. The standard for chlordane has peaks that can interfere with other pesticides and thus complicate the interpretation of the chromatogram. Table 4 indicates the presence of dieldrin for several samples, but the concentration of this compound was in trace amounts in most cases. Quantitation of the observed compounds was not performed.

The procedures presented here can serve as a starting point for future research concerning pesticides in solid waste. When analyzing for pesticides with these procedures in the future, however, the literature should be further screened for TLC systems and solvent systems for extracting pesticides.

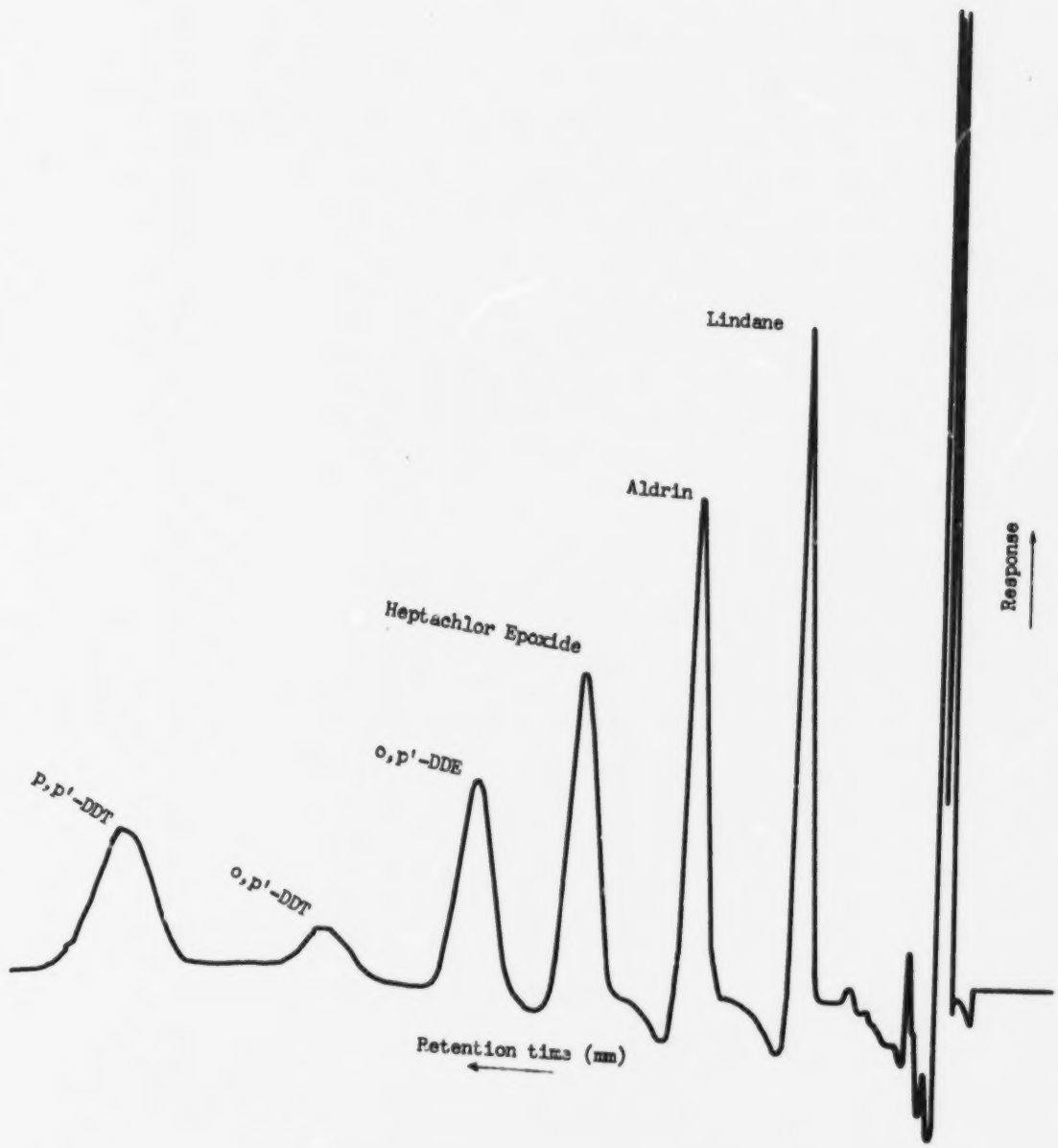


Figure 2. Chromatogram of mixed pesticide standards using Instrument B. Final concentration was 0.2 ng for lindane, aldrin, heptachlor epoxide, and o,p'-DDE; 0.14 ng for o,p'-DDT; and 0.64 ng for p,p'-DDT. (Reduced 20%.)

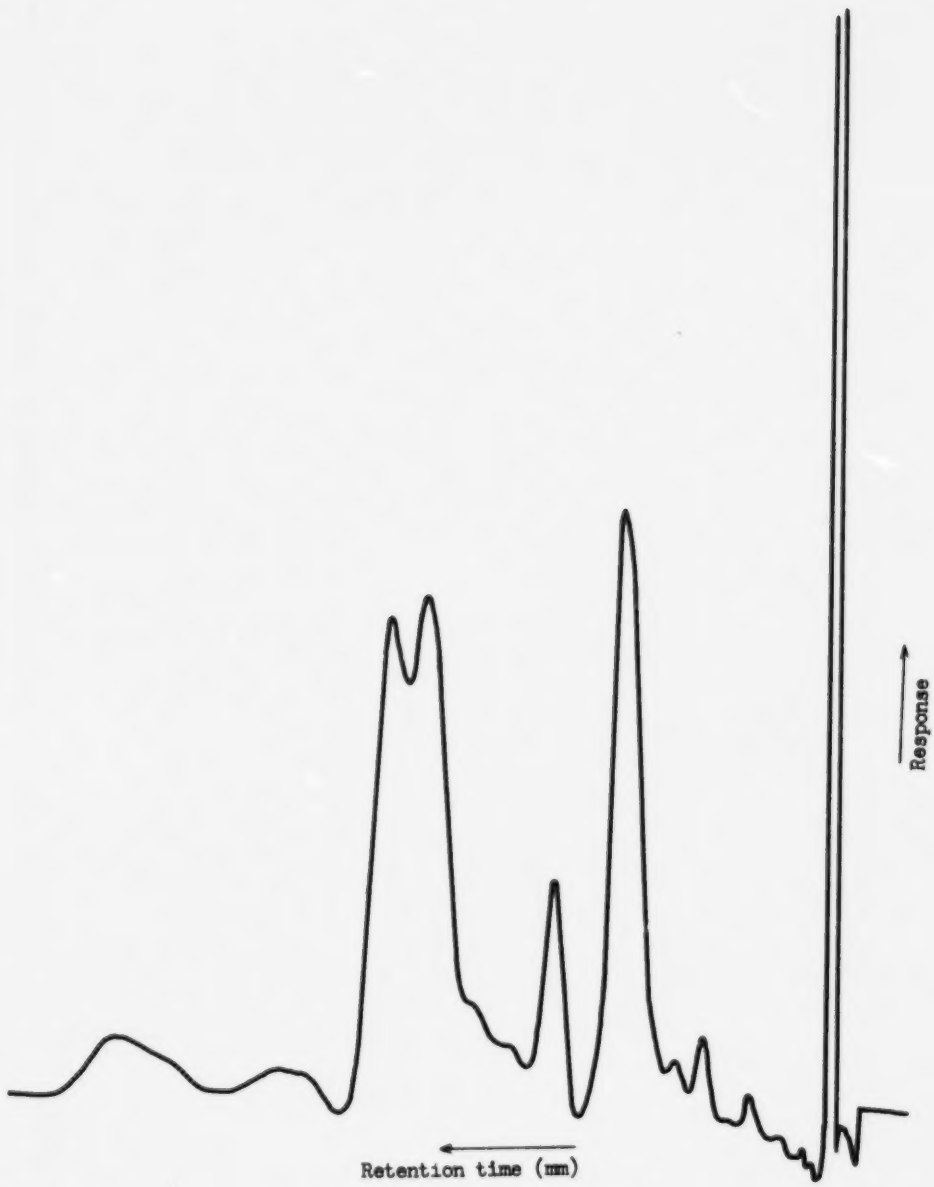


Figure 3. Chromatogram of 5 ng chlordane standard from Instrument B. (Reduced 20%.)

Figure 4. Chromatogram of 0-day compost from Instrument B showing evidence of lindane, chlordane (see multiple peaks in Fig. 3), and p,p'-DDT. (Reduced 20%.)

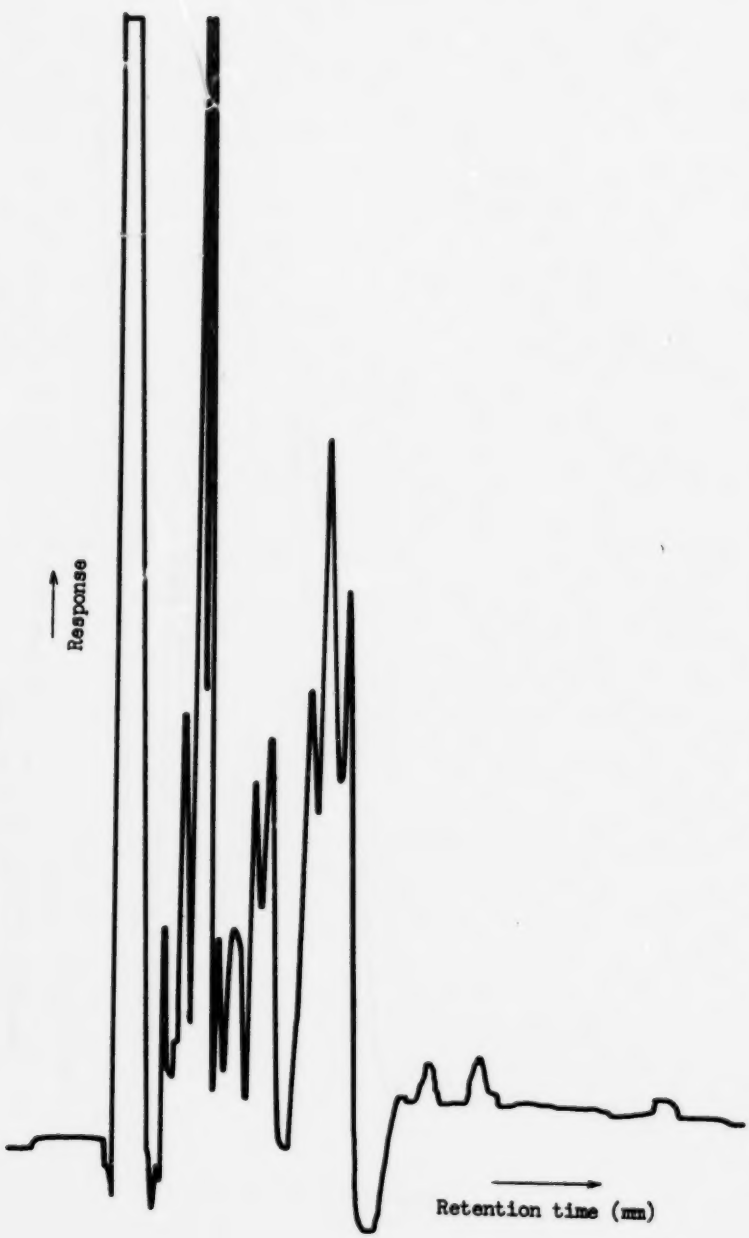


Figure 5. Chromatogram of 0-day compost from Instrument C showing evidence of lindane, p,p'-DDE, o,p'-DDE, p,p'-DDT, chlordane. See Table 2. (Reduced 20%.)

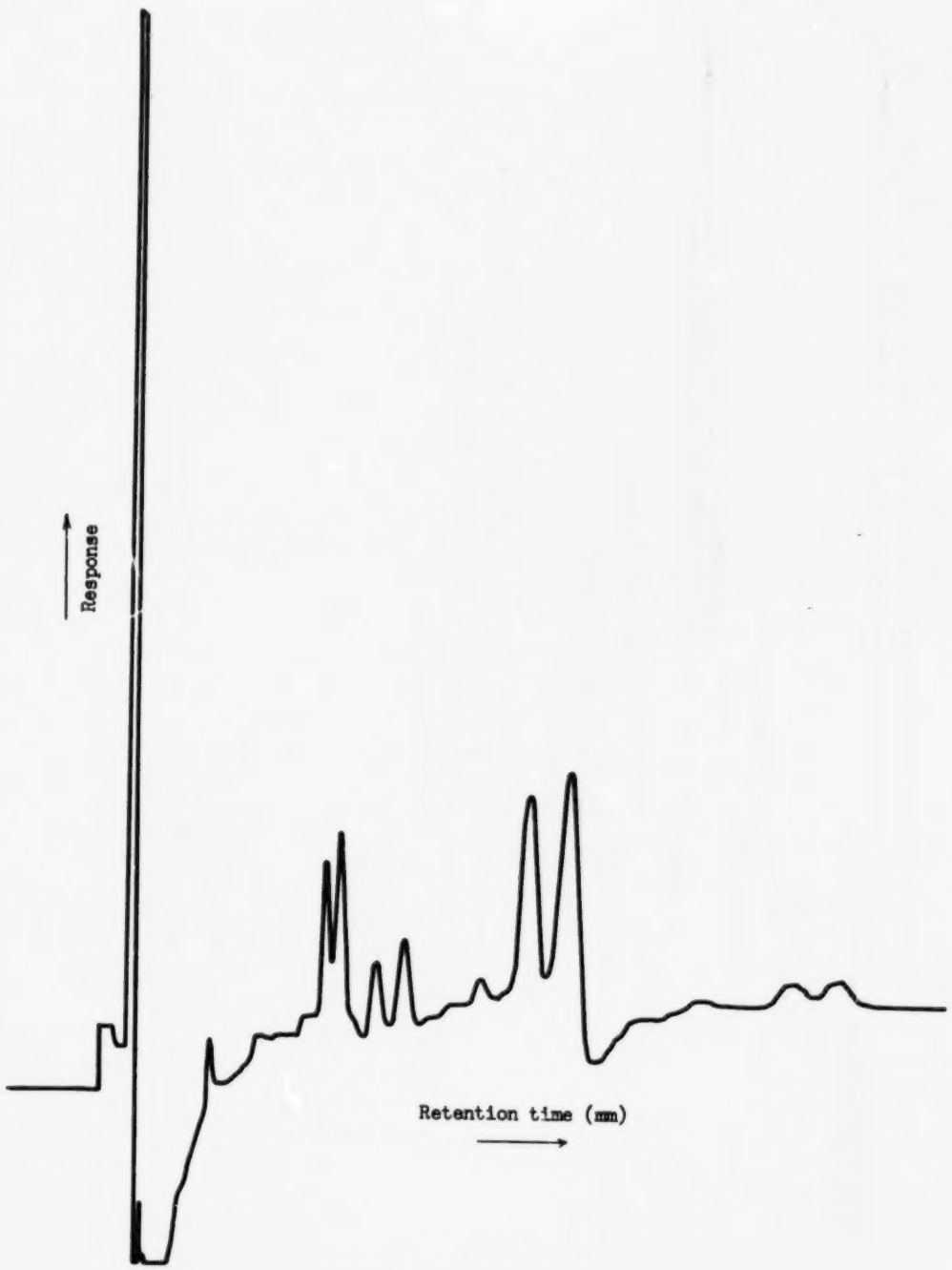


Figure 6. Chromatogram of chlordane standard from Instrument D. (Reduced 20%.)

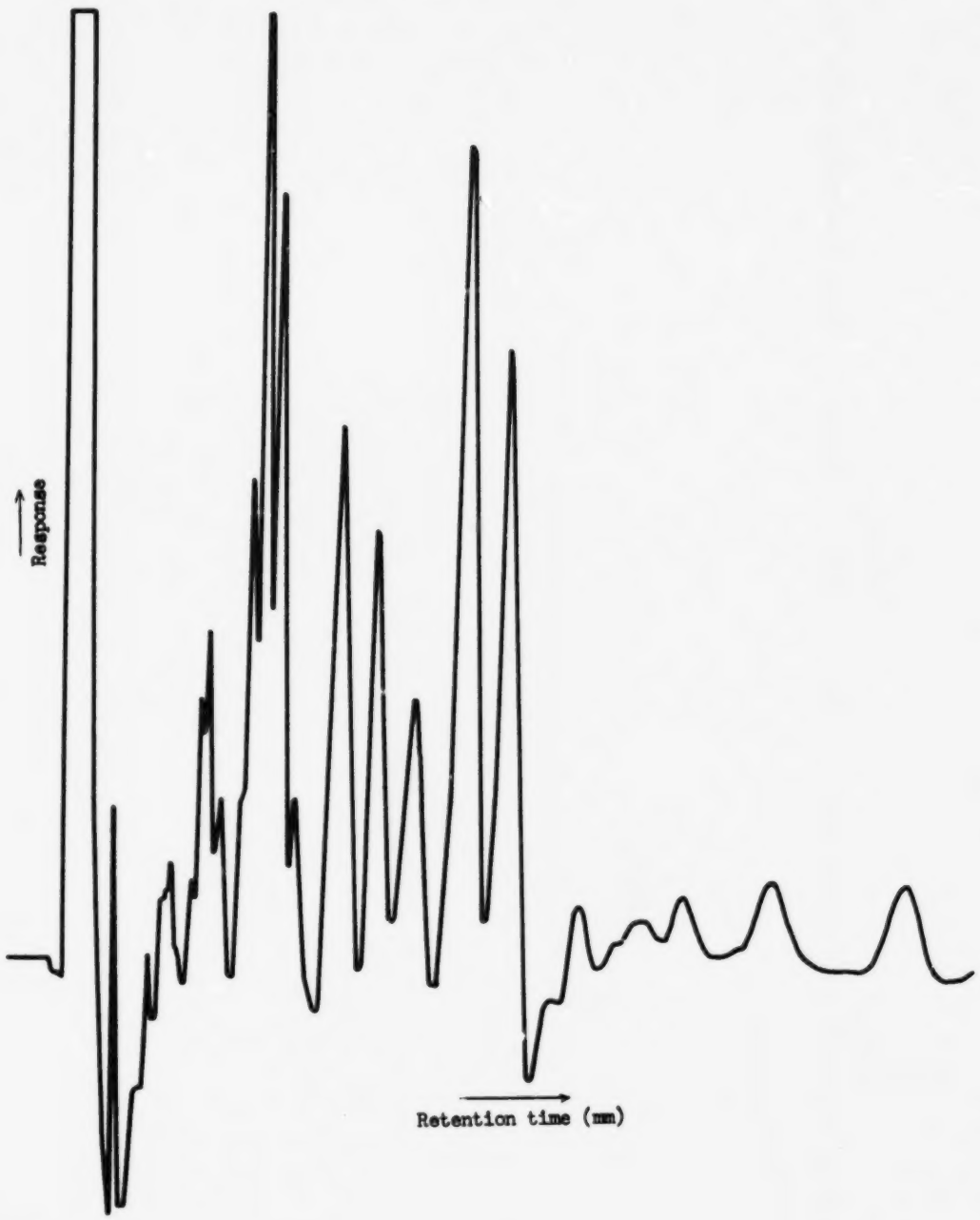


Figure 7. Chromatogram of 0-day compost from Instrument D showing evidence of lindane, o,p'-DDD, p,p'-DDT, and chlordane. See Table 2. (Reduced 20%.)

TABLE 2. R_A* VALUES FOR PESTICIDE STANDARDS AND SAMPLES

Standard and samples	Pesticide: Lindane			o,p'-DDE			p,p'-DDE		
	Instrument: B	C	D	B	C	D	B	C	D
Standard	0.58	0.68	0.57	1.85	1.91	1.53	---	2.36	1.83
Sample 1A†					1.91				1.89
2A					1.91	1.51			1.84
3A									
4A				1.85	1.94	1.50		2.36	1.83
5A				1.85		1.51			1.82
6A					1.91			2.36	1.82
7A									
8A	0.58	0.69	0.57		1.94			2.36	
9A					1.94	1.51			

Standard and samples	Pesticide: o,p'-DDD			p,p'-DDT				Dieldrin		
	Instrument: B	C	D	B	C	D		B	C	D
Standard	---	2.84	1.98	3.22	4.55	3.19		2.15	2.54	2.16
Sample 1A†							1B††	2.16	2.53	2.15
2A			1.94				2B			2.16
3A			1.98				3B	2.15		2.17
4A				3.22	4.55	3.19	4B	2.15	2.52	2.17
5A			1.96				5B	2.15		2.19
6A			1.94	3.17		3.15	6B			
7A			1.98				7B			2.20
8A			1.94	3.18	4.55	3.26	8B		2.54	2.14
9A			1.94				9B		2.52	2.19

*R_A, Relative Retention Time (relative to aldrin).

†From 6% fraction.

††From 15% fraction.

TABLE 3. PESTICIDES INDICATED FROM THE 6% FRACTION* BY VARIOUS INSTRUMENTS†

	Instrument		
Sample	B	C‡	D
1A	chlordane	o,p'-DDE (trace)	chlordane, p,p'-DDE
2A	chlordane (trace)	o,p'-DDE (trace)	p,p'-DDE, o,p'-DDD, o,p'-DDE
3A	----	----	o,p'-DDD, chlordane
4A	chlordane, p,p'-DDT o,p'-DDE	p,p'-DDT, o,p'-DDE p,p,-DDE	chlordane, p,p'-DDT, o,p'-DDE
5A	chlordane, o,p'-DDE	chlordane	chlordane, o,p'-DDD p,p'-DDE
6A	chlordane	o,p'-DDE (trace) p,p'-DDE	chlordane, p,p'-DDE o,p'-DDD, p,p'-DDT
7A	chlordane	----	chlordane, o,p'-DDD
8A	chlordane, lindane p,p'-DDT	lindane p,p'-DDE, p,p'-DDT o,p'-DDE	chlordane, o,p'-DDD p,p'-DDT, lindane
9A	----	DDA(ME) [possible]	o,p'-DDD (trace) o,p'-DDE

*First eluate with 200 ml of 6% ethyl ether in hexane.

†The R_A values did not correspond for instrument D as well as they did for instruments B and C. Therefore, there remains some doubt as to positive identification.

‡No chlordane standard run on this instrument.

TABLE 4. PESTICIDES INDICATED FROM THE 15% FRACTION* BY VARIOUS INSTRUMENTS

	Instrument		
Sample	B	C	D
1B	dieldrin	----	dieldrin
2B	----	----	dieldrin
3B	dieldrin (trace)	----	dieldrin
4B	dieldrin	----	dieldrin
5B	dieldrin (trace)	----	dieldrin
6B	----	----	----
7B	----	----	----
8B	----	dieldrin	dieldrin
9B	----	----	dieldrin

*Second eluate with 200 ml of 15% ethyl ether in hexane.

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